MATERIALS AND FABRICATION METHODS FOR HIGH TEMPERATURE MICRO-MAGNETIC MACHINES FOR MICRO-TURBINE POWER GENERATION

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ABSTRACT

This paper[1] presents recent advances in the development of materials and fabrication methods for high temperature (400 - 900 °C) magnetic micro-machine for electrical power generation. Suitable materials and material compatibility issues are identified for the device conductors, magnetic cores, and bulk structure. Two fabrication methods for encapsulating electroplated structures inside fusion bonded silicon wafers are presented. These fabrication techniques enable the integration of thick conductive and magnetic materials into multi-wafer silicon MEMS devices. Electrical tests verify the integrity of electroplated Cu conductors after wafer bond annealing and mechanical tests confirm that the presence of the metal does not adversely affect the bond strength. Preliminary results of electrodeposited ferromagnetic Fe-Co materials are also discussed.

INTRODUCTION

Army concepts such as the Objective Force Warrior will require energy sources with many times the energy and power density of today's best batteries. This drives the development of compact electric power sources in the 10-100 watt range, suitable for use by soldiers, unattended sensors, or robotic devices. One potential system is a micro turbine generator—a small (few cubic centimeters) turbine engine coupled to an electrical generator running on a hydrocarbon fuel. Such a system would reduce the mass of soldier power systems, eliminate the cumbersome logistics of batteries, and reduce the life-cycle cost of soldier power units.

Progress has been made on the development a multi-wafer silicon micro-turbine to convert fuel into mechanical energy [1,2]. An electrical machine is needed to convert the mechanical energy from the turbine into electrical power. One approach is the use of a magnetic induction machine. We have previously reported the construction of a two-phase linear induction micro-machine using micro-molding and electroplating of various metals [3]. High aspect ratio, ultra thick, photosensitive materials such as SU-8 epoxy enabled the fabrication of voluminous MEMS magnetic structures (mm thick, hundreds of microns across) as shown in Figure 1. This first-generation device was not directly integrable with the silicon-based micro-turbine and its operating temperature was limited to 300°C due to the presence of the SU-8 polymer. However, the device verified the machine design and successfully demonstrated mechanical-electrical transduction. This paper investigates high temperature materials and proposes and validates fabrication methods that will enable direct integration with the silicon micro-turbine.

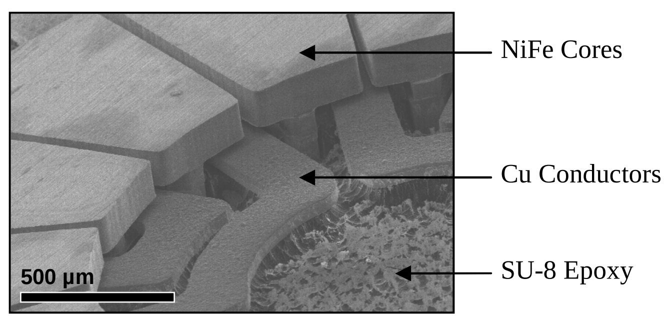

Figure 1. SEM of previously reported low temperature magnetic induction machine [3]. The SU-8 mold is partially removed to show the top Cu coil wound in between the "T"-shaped NiFe stators.

HIGH TEMPERATURE MATERIALS

Three classes of materials must be investigated for use in the micro generator as shown in Figure 2. To be

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[1] Prepared through collaborative participation in the Power and Energy Consortium sponsored by the U. S. Army Research Laboratory under the Collaborative Technology Alliance Program, Cooperative Agreement DAAD19-01-2-0010. The U. S. Government is authorized to reproduce and distribute reprints for Government purposes notwithstanding any copyright notation thereon.

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE **2003**	2. REPORT TYPE	3. DATES COVERED **00-00-2003 to 00-00-2003**
4. TITLE AND SUBTITLE **Materials and Fabrication Methods for High Temperature Micro-Magnetic Machines for Micro-Turbine Power Generation**		5a. CONTRACT NUMBER 5b. GRANT NUMBER 5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)		5d. PROJECT NUMBER 5e. TASK NUMBER 5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **Georgia Institute of Technology,School of Electrical and Computer Engineering,Atlanta,GA,30332**		8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSOR/MONITOR'S ACRONYM(S) 11. SPONSOR/MONITOR'S REPORT NUMBER(S)
12. DISTRIBUTION/AVAILABILITY STATEMENT **Approved for public release; distribution unlimited**		
13. SUPPLEMENTARY NOTES		
14. ABSTRACT		
15. SUBJECT TERMS		

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**		**5**	

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std Z39-18

compatible with the micro-turbine, these materials must withstand the high temperatures required for direct silicon bonding and sustained operation at several hundred degrees Celsius. Oxidation, diffusion, and thermal mismatch must also be addressed.

Material	Purpose	Desired Properties
Conductor	Conduct current in coils.	-low resistivity -no electromigration
Core	Guide flux in magnetic core.	-high flux density -high Curie temp. -high resistivity
Structural	Support and isolate coils and cores.	-good electrical insulator -high aspect ratio -high heat conduction

Figure 2. Materials required for a micro-magnetic induction machine.

Silicon is an obvious choice to serve as the structural material. Direct silicon (fusion) bonding can be used to directly integrate the electrical generator in the fabrication of the turbine. Silicon can be patterned with extremely high precision using deep reactive ion etching (DRIE) techniques and can also be oxidized to provide electrical isolation. The melting points of many electroplatable metals are relatively high (Cu, Ni, Co, and Fe are 1083, 1453, 1495, and 1535 °C, respectively) as is Si (1414 °C). Therefore, electroplating techniques remain an appropriate way to deposit thick conductors and ferromagnetic materials and will continue to serve in the construction of a high temperature device. Sacrificial polymers can be used to form molds for the electroplated structures, but permanent polymers should be avoided due to the low decomposition temperatures of most polymers.

Copper is selected as the conductor material for its low resistivity, high resistance to electromigration, and relatively high melting point. However, Cu readily reacts with Si to form Cu-Si compounds at temperatures as low as 200°C, thus necessitating a diffusion barrier such as Ta or TaN [4]. To prevent oxidation and corrosion during operation at elevated temperatures, copper can be over-plated with a more stable metal such as nickel or platinum.

High temperature magnetic materials are crucial for the success of the magnetic device. Almost no known permanent magnetic materials can operate beyond 500 °C [5,6]. This is not the case soft magnetic materials. Although previously used NiFe electroplated alloys exhibit a Curie temperature below 300 °C, Fe-Co alloys at 50%-50% composition (Permendur) exhibit high Curie temperature (940 °C), high relative permeability (5000), and high saturation magnetization (1.9 - 2.2 T) but depending on the composition, such alloys are extremely brittle [7]. Other elements such as vanadium (V) are usually added to obtain workable materials and can increase the electrical resistivity and thus reduce eddy current losses. Various studies have been reported for methods for electrodeposition of Fe-Co alloys [8,9]. The composition of the plating bath can be tailored to vary the composition of the deposited material and annealing may be required to achieve maximal magnetic properties [9].

FABRICATION METHODS

Two fabrication methods were developed to integrate electroplated metals with silicon wafer bonding as shown in Figure 3. In the first method, metal is electroplated on a flat wafer, which is then bonded to another wafer with corresponding cavities. In the second method, metal is plated inside pre-etched cavities and a flat wafer is then bonded to seal the cavities.

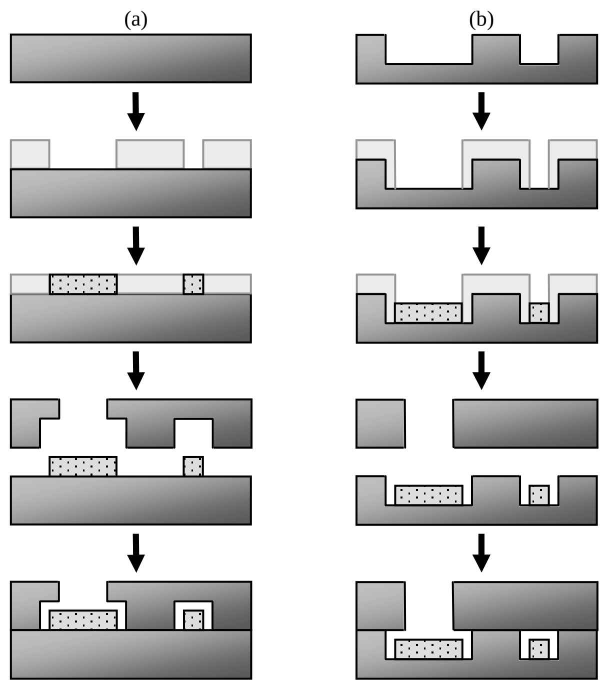

Figure 3. Two methods for embedding electroplated metal structures in wafer bonded silicon, depicting a bond pad and an encapsulated line: (a) structures plated on a flat wafer and then bonded to a wafer with cavities; (b) structures plated in cavities and then bonded to a flat wafer.

The general fabrication sequence begins by growing 1 μm thermal oxide on a flat or pre-etched (~125 μm) silicon wafer. A diffusion barrier, followed by a conductive seed layer are sputtered across the entire wafer. The diffusion barrier prevents interaction of the metal with the silicon and the seed layer is needed for electrodeposition. Futurrex NR9-8000 negative photoresist is patterned to

define the electroplating mold. In the case of the pre-etched wafers, the pattern is defined down within the trench. The metal is then electroplated to the desired thickness (25-100 µm). The mold is stripped and the non-plated areas are etched back down to the oxide surface. The oxide is wet-etched to yield a pristine silicon surface and the wafers are cleaned, aligned, bonded, and annealed.

EXPERIMENTAL

Embedded electroplated copper test structures were fabricated at Georgia Institute of Technology using both methods described above. Copper was plated on a 1 µm oxide with a 2000 Å Ta diffusion barrier and 2000 Å Cu seed layer using a conventional Cu plating bath as listed in Figure 4. The test structure lines were 35 µm thick and varied from 50 – 200 µm wide. The samples were then bonded and annealed at temperatures ranging from 500 – 900 °C for 1 hr. An example of the test structures before and after wafer bonding is shown in Figure 5. Figure 6 shows cross-sections of encapsulated copper lines. Potential high-temperature magnetic materials were also investigated. Fe-Co test structures from various plating bath chemistries were electroplated into micro-molds on flat substrates and preliminary characterizations were performed.

Component		Units
$CuSO_4 \cdot 5H_2O$	250	g/L
H_2SO_4	25	mL/L
Temperature	25 – 30	°C
Current density	10	mA/cm^2
Anode	Copper	

Figure 4. Bath chemistry for electrodeposition of Cu.

<u>Electrical Resistance Tests</u>

To determine the thermal bounds for the wafer-bond annealing step, four-point electrical resistance measurements were made on copper structures before and after 1 hr. thermal cycles. The heat treatments were performed in a tube furnace under nitrogen ambient, simulating the conditions for wafer bond annealing. The resistance of the test structure was measured by passing a current of 1 A through the test structure and measuring the corresponding voltage as shown in Figure 7. Figure 8 shows the change in resistance after 1 hr. thermal cycles at increasing temperatures. The electroplated copper exhibits good adhesion and nearly constant resistance at temperatures up to 900°C. An overall slight decrease in temperature is observed due to a sintering effect in the electroplated metal. Fully encapsulated structures have been shown to maintain resistance after annealing at 500°C for 1 hr.

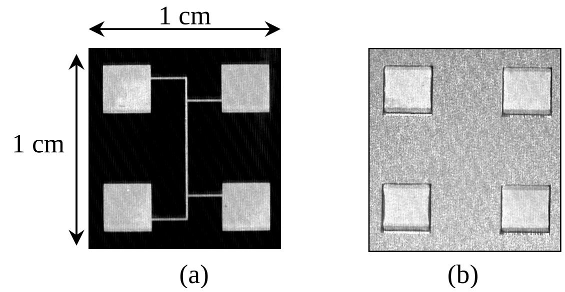

Figure 5. Electroplated copper test structure (a) before bonding and (b) after bonding showing the bond pads.

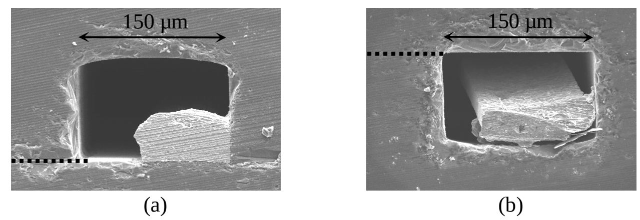

Figure 6. Cross sectional SEMs show examples of encapsulated copper lines fabricated by the two methods. The dashed line indicates the location of the bond interface. Some dicing saw artifacts can be seen.

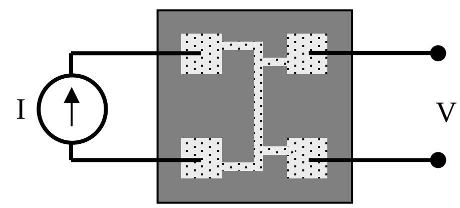

Figure 7. Diagram of setup for four-point resistance test.

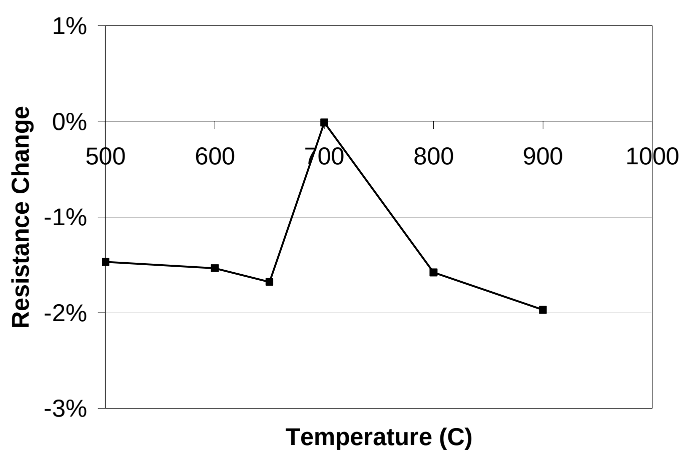

Figure 8. Average resistance change of electroplated Cu on 2000Å Ta/1 µm SiO_2/Si substrate after annealing in nitrogen at various temperatures for 1 hr.

Mechanical Tests

Tensile tests were conducted at Clark Atlanta University to verify that the presence of the electroplated metal does not adversely affect the quality of the bond. Bonded samples were constructed with cavities either with or without copper present. The bonded samples were mounted to steel block test fixtures using cyanoacrylate adhesive and tensile loads were applied using an MTS loading frame as shown in Figure 9. Samples that included copper exhibited bond strengths greater than or equal to samples bonded with empty cavities for several different annealing conditions as shown in Figure 10. It seems evident that the presence of copper does not reduce the bond strength.

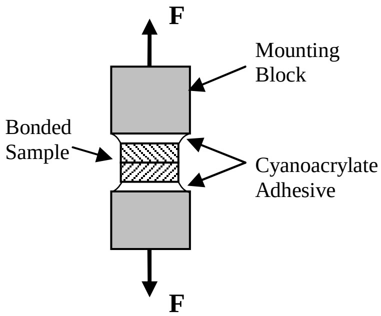

Figure 9. Method for tensile testing wafer bonded pairs, depicting the bonded sample mounted to the mounting blocks with cyanoacrylate. The de-bonding force is measured under an applied load.

Anneal	With Cu	Without Cu
None	3.7 MPa	--
500°C, 1 hr.	5.3 MPa	4.3 MPa
900°C, 1 hr.	12.2 MPa	6.6 MPa

Figure 10. Comparison of average de-bonding failure force for wafer bonded samples with and without Cu present.

Magnetic Tests

We initiated investigations into potential high temperature electroplated Fe-Co materials using two different baths based on $FeSO_4$ and $CoSO_4$ as listed in Figure 11. Bath 1 was reported by Park and Allen and contained Cu to reduce the brittleness [10]. FeCoCu structures were plated 15 μm thick in 1.5 hr. and EDS analysis revealed a 12%-80%-8% composition. Preliminary electrical measurements indicate a resistivity of approximately 50 μΩ·cm. Magnetic tests indicated a saturation magnetization of 0.6 T and a relative permeability of 73, much lower than expected. Annealing steps may improve the magnetic properties.

Bath 2 was a modified version to eliminate the Cu and balance the composition of FeCo to 50%-50%. The composition of the deposited alloy was 58%-42% but the brittleness of the material indicated an extra element may be necessary. No electrical or magnetic characterization was performed on samples from Bath 2.

A second bath chemistry based on $FeCl_2$ and $CoCl_2$ was investigated as listed Figure 12. This bath is slightly modified from Paunovic and Shlesinger, again to balance the composition of Fe and Co [11]. The bath seemed to etch away much of the Cu/Ti seed layer resulting in a flaky, brittle deposition. This etching is attributed to the presence of chloride and fluoride ions in the bath. The plated alloy composition is very close to 50%-50% but the brittle nature of the alloy indicates once again that the plating conditions or the bath chemistry and composition should be altered. No electrical or magnetic tests were performed on samples from Bath 3.

Component	Bath 1	Bath 2	Units
$CH_3COONa \cdot 3H_2O$	11.05	11.05	g/L
$CoSO_4 \cdot 7H_2O$	56.2	30	g/L
$FeSO_4 \cdot 7H_2O$	8	30	g/L
$CuSO_4 \cdot 5H_2O$	0.5	0	g/L
H_3BO_3	24.7	24.7	g/L
Saccharin	3	3	g/L
pH	2.5 – 4	2.5 – 4	
Temperature	25 – 30	25 – 30	°C
Current density	7	7	mA/cm^2
Anode	Cobalt	Cobalt	

Figure 11. Fe-Co bath chemistries based on $FeSO_4$ and $CoSO_4$. Bath 1 was taken from Park and Allen [10].

Component	Bath 3	Units
$FeCl_2 \cdot 4H_2O$	60	g/L
$CoCl_2 \cdot 6H_2O$	50	g/L
H_3BO_3	10	g/L
NH_4Cl	75	g/L
NaCl	75	g/L
NH_4BF_4	10	g/L
Temperature	25 – 30	°C
Current density	50	mA/cm^2
Anode	Cobalt	

Figure 12. Fe-Co bath based on FeCl2 and CoCl2. Modified from Paunovic and Schlesinger [11].

SUMMARY & CONCLUSIONS[2]

A range of experiments were conducted in order to identify materials and fabrication steps needed for the fabrication of high temperature magnetic machine. Copper test structures were used to demonstrate that electroplated metals can be incorporated into wafer bonded silicon devices. The methods presented are suitable for a variety of electroplated materials in silicon and are well suited for magnetic MEMS devices.

Electroplated Cu was shown to maintain constant resistance at temperatures up to 900°C for 1 hr using a 2000Å Ta diffusion barrier. Wafer bonded structures with encapsulated metals were shown to be as strong as bonded structures without metal. The plating of Fe-Co was demonstrated and variations on the bath chemistries were shown to modify the composition of the deposited alloy.

Work is underway to extend the limits of the wafer bond annealing to improve the bond strengths. Alternative molding techniques using various sacrificial polymers are being investigated to increase the aspect ratio and improve the yield of the plated structures. Once a plating bath is found that yields Fe-Co with reasonable mechanical properties, electrical and magnetic characterizations will be performed. Various annealing cycles will be explored as ways to improve the magnetic properties of the Fe-Co structures. Future work will aim at measuring the bond strengths and electrical and magnetic properties in situ at elevated temperatures.

ACKNOWLEDGEMENTS

This work was supported by the Army Research Office under contract DAAG55-98-1-0292 managed by Dr. T. L. Doligalski, and a contract from the Army Research Laboratory managed by Mr. J. Hopkins. Appreciation is extended to Brian Shonkwiler at Clark Atlanta University for his assistance in the mechanical testing.

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